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NASA TN D-2329

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Lewis Research Center

Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • JUNE 1964

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SUMMARY

The bombardment of sodium chloride crystals with high fluxes ($>10^{14}$ particles/cm^2) of 40-Mev alpha particles, whose computed range is 0.72 millimeter, produces a damaged planar region normal to the alpha beam near the end of the range. The irradiated crystals are colored throughout the portion between the incident surface and a plane parallel to this surface at a depth of 0.73 ± 0.01 millimeter. The damaged region is characterized by (1) a line of discontinuity of the surface cleavage pattern at the coloration limit on surfaces parallel to the beam, (2) a plane of "easy" cleavage at various depths between 0.43 and 0.62 millimeter, and (3) crystallographic cavities that appear after the crystals are heated to 400° C. These cavities form in planes at the coloration limit, at the easy cleavage plane, and sometimes in planes at slightly shallower depths. The heating suffices to bleach completely the F-centers produced by the irradiation, and, during this time, the helium gas from the stopped alpha particles escapes from the crystals. Chemical analyses show that some "free" chlorine and "free" sodium atoms are present in the irradiated colored crystals, but that the irradiated bleached crystals contain no free chlorine or sodium.

INTRODUCTION

During the course of an investigation of the thermoluminescence of sodium chloride irradiated with 40-Mev alpha particles (ref. 1), it was observed that the heavy radiation damage produced a region containing planes of "easy" cleavage near the end of the particle range (ref. 2). These planes were normal to the incident-beam direction and were such that the crystal would cleave at one of these planes when tapped lightly with a small hammer. The irradiated crystals that were subsequently heated and bleached showed a macroscopic light-scattering plane in the same region. Microscopic examination of this plane revealed that the light-scattering phenomenon was due to parallelepipedal cavities (most of which were cubic and are hereinafter referred to as crystallographic cavities). These proved to be similar in appearance to the "bubbles" found in alpha-particle-irradiated copper (ref. 3) and to the "cavities" found in neutron-irradiated lithium fluoride (refs. 4 to 6).

The investigations of the damage in these other crystals led to the proposal of various mechanisms to account for the cavity formation. Among these were proposals that, during annealing, helium gas atoms precipitate in the form of gas bubbles, and, in the case of lithium fluoride, the cavities are caused by loss of fluorine. Because the microscopic character of the sodium chloride cavities was similar to those of lithium fluoride and copper, and because the cavities were formed under somewhat similar circumstances, (namely, from bombardment with either fission alpha particles or machine-accelerated alpha particles) an attempt was made to ascertain if either of the proposed mechanisms for cavity formation in lithium fluoride or copper could explain the cavities observed in the present sodium chloride specimens. As part of this program, both irradiated colored crystals and irradiated bleached crystals were measured for helium content, "free" chlorine content, and "free" sodium content. Photomicrographs were made of both colored and bleached crystal specimens that had been bombarded with fluxes of 10^{15} and 5×10^{15} alpha particles per square centimeter. The higher flux gave about the same amount of ionization as that produced in a similar lithium chloride crystal bombarded with 10^{18} neutrons per square centimeter.

The measurements of free sodium and free chlorine contents were made by D. Otterson of Lewis.

EXPERIMENTAL PROCEDURE

The crystals used in this study were single crystals of optical grade sodium chloride (obtained commerically with traces of O_2-bearing anions such as OH^-, etc.) that were cleaved to about 20 by 20 by 2 millimeters, annealed at 550° C for 1 hour in vacuum, then cooled to room temperature at a rate of 25° C per hour, and irradiated in a liquid-nitrogen Dewar (ref. 1) with 40-Mev alpha particles at a beam current of 1 microampere. After the bombardment that gave integrated fluxes of 10^{15} and 5×10^{15} particles per square centimeter, the crystals were stored in liquid nitrogen in the dark for 15 to 20 hours, while the radioactivity decayed to a safe handling level. Prior to thermoluminescence measurements, the crystals were removed from the nitrogen, permitted to reach room temperature in a dry atmosphere, and cleaved into specimens 4 by 5 by 2 millimeters. Some of the specimens were bleached during the course of thermoluminescence measurements by being heated at a rate of 0.9° C per second to 400° C. At 400° C, the furnace was turned off, and the crystals were allowed to cool to room temperature over a period of about 10 minutes. Bleaching was done and examination was made within 3 days after the bombardment.

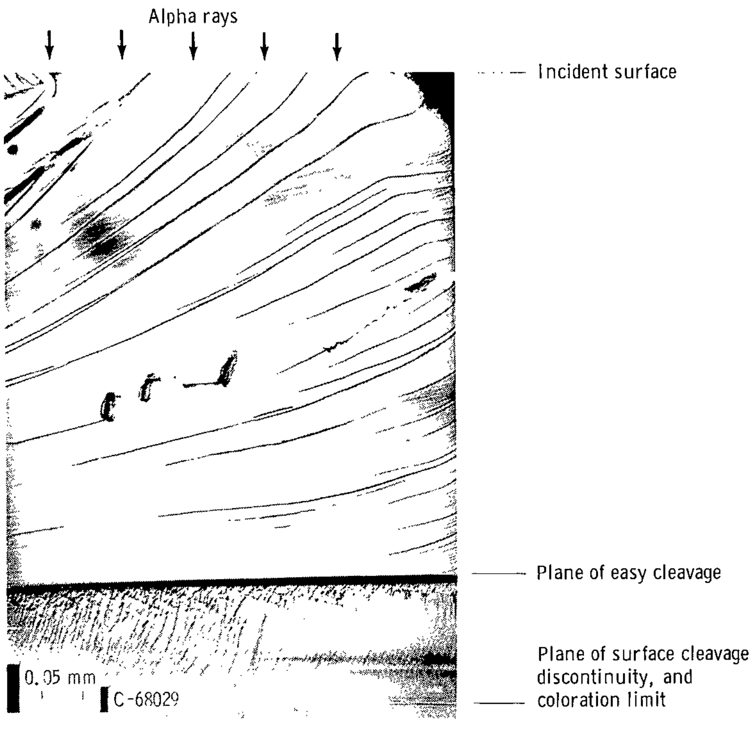
A crystal irradiated with an integrated flux of 5×10^{15} alpha particles per square centimeter was cleaved into 10 samples that were checked for the presence of helium. Prior to this check, one-half of the samples were heated until they were bleached, and then all were checked in the following manner. The samples were placed inside a vacuum chamber connected to a helium leak detector and then crushed to release any helium from the alpha-particle bombardment. The detector can sense a minimum flow of helium of about 5×10^{12} atoms per second. The samples of frontal area of 0.20 square centimeter contained about 10^{15} helium atoms. If all this helium were released when the samples were crushed, there would be a readily detectable flow of helium.

The amounts of free chlorine and free sodium present in the irradiated colored crystals and in the irradiated bleached crystals were measured in samples taken from two separately bombarded crystals. Six samples were used in each set of measurements. The free chlorine was measured colorimetrically from the reaction of ortho-tolidine and the free chlorine of the crystal in an aqueous solution (ref. 7). Similarly, the amount of free sodium present was measured from the reaction of para-nitrophenol and the free sodium of the bombarded crystal in aqueous solution (ref. 8). The accuracy of the measurement was $\pm 1\times10^{15}$ atoms per cubic centimeter for free chlorine, and $\pm 4\times10^{16}$ atoms per cubic centimeter for free sodium.

RESULTS

Visual Observations

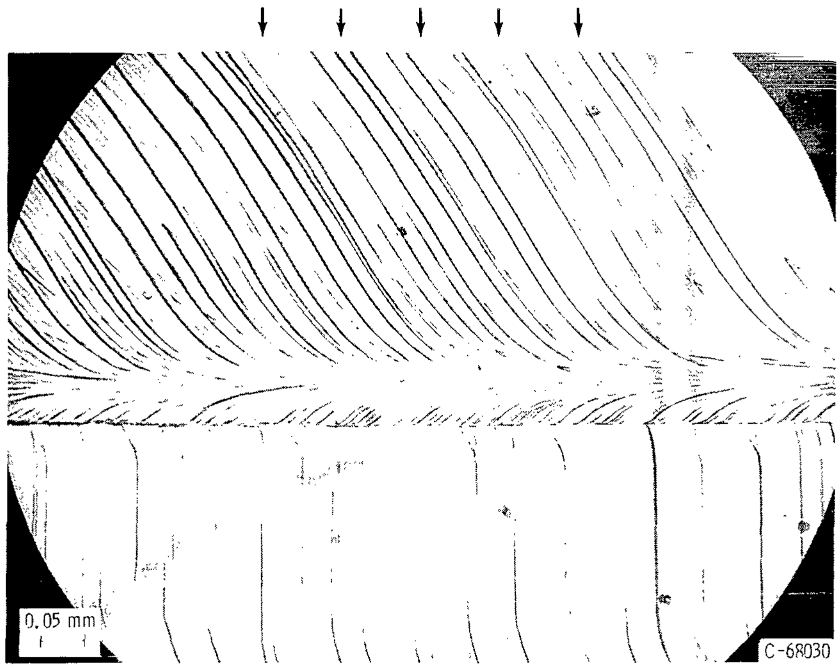
Microscopic observations and photomicrographs were made of the cubic faces of the crystal at magnifications of 65, 135, and 335. Figure 1 shows photomicrographs of the (100) plane of specimens that were irradiated but not subsequently heated. The crystals were bombarded with a total flux of 5×10^{15} particles per square centimeter. Three effects of the particle irradiation on the crystals may be noted. The first is the production of color centers in the crystals; the 40-Mev alpha particles are stopped in the crystal and produce a colored region 0.73±0.01 millimeter in depth. The second effect is the discontinuity of the surface cleavage pattern at the limit of the colored region. The third effect is the existence of a plane of easy cleavage, that is, a plane at which the crystal will separate when it is tapped with a blunt object. This plane of easy cleavage is visible in figure 1(a) as a surface crack extending horizontally across the picture (the surface crack in this crystal did not extend across the entire crystal). In figure 1(b), the plane of easy cleavage is manifested by a sharp but continuous 45° change in the direction of the surface cleavage pattern. The lines of surface separation and the sharp change in surface cleavage pattern usually are present at this plane.



(a) Surface crack extending horizontally across crystal.

Figure 1. - Photomicrographs of (100) plane of crystal specimens.

Alpha rays



(b) Continuous 45° change in direction of surface cleavage pattern.

Figure I. - Concluded. Photomicrographs of (100) plane of crystal specimen.

The easy cleavage plane is accompanied by a surface fracture in the crystals irradiated with heavier dosages, that is, with fluxes greater than 10^{15} alpha particles per square centimeter. This plane is not uniquely positioned but appears randomly between extreme depths of 0.43 and 0.62 millimeter with an average depth of 0.54±0.04 millimeter. This position is in about the last one-third of the particle range where the crystal is most heavily damaged (as a result of the nonuniform radiation damage along the beam path).

When the crystals are heated to 400° C, the F-centers are bleached, and a light-scattering plane appears in the crystal. Figure 2 shows this plane in a typical specimen; the plane is normal to the incident beam and at a depth corresponding to the plane of easy cleavage. The structure of the light-scattering plane can be seen in figure 3, which shows two halves of a crystal that was cleaved normal to the damage plane after being irradiated. The left half was not heated (fig. 3(a)). The right half is shown after heating to 400° C (fig. 3(b)). In figure 3(a) the terminal portion of a surface fracture at the plane of easy cleavage is shown in the middle at the right side of the photograph. The discontinuity of the surface cleavage pattern and the end of the coloration are shown at the bottom of the figure. (A diagonal flake somewhat obscures the surface.) The corresponding surface of the right half of the crystal after bleaching (fig. 3(b)) shows a band of crystallographic cavities (bubbles) in planar regions normal to the incident alpha beam. Although poorly defined, three separate planar regions that contain cavities can be discerned. (These regions are shown much more clearly in fig. 4.) The region corresponds to the position of the coloration limit and the surface cleavage pattern dis-

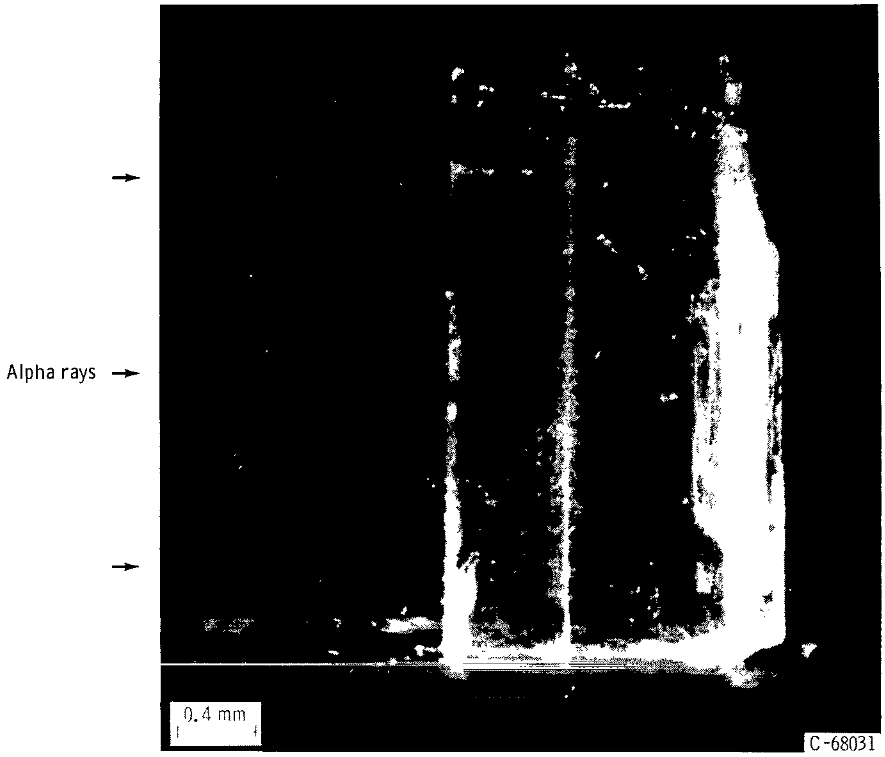
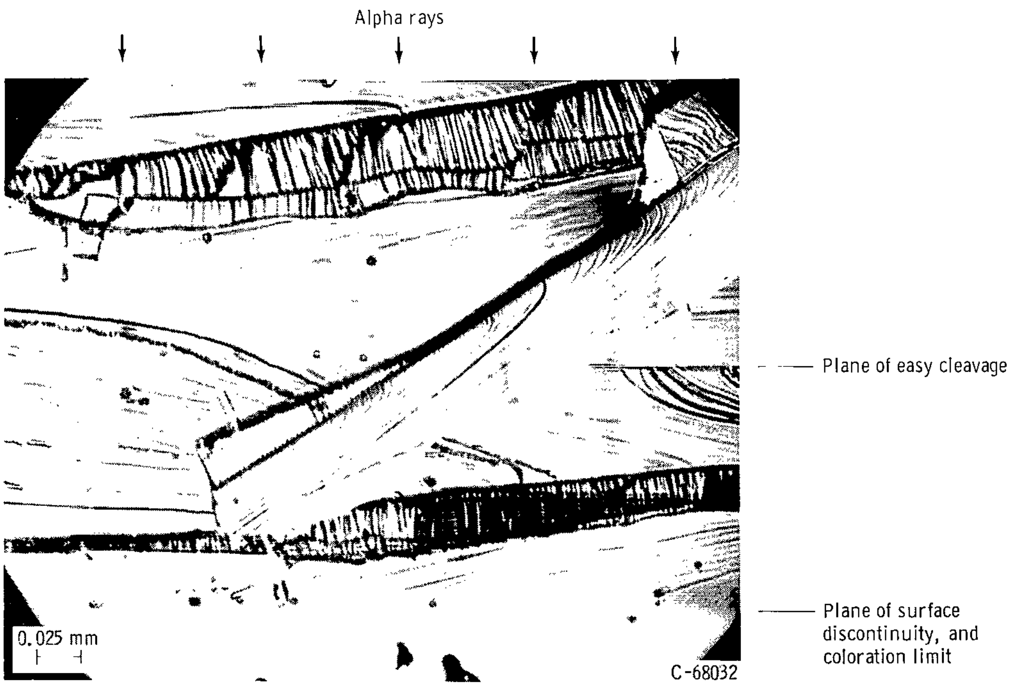
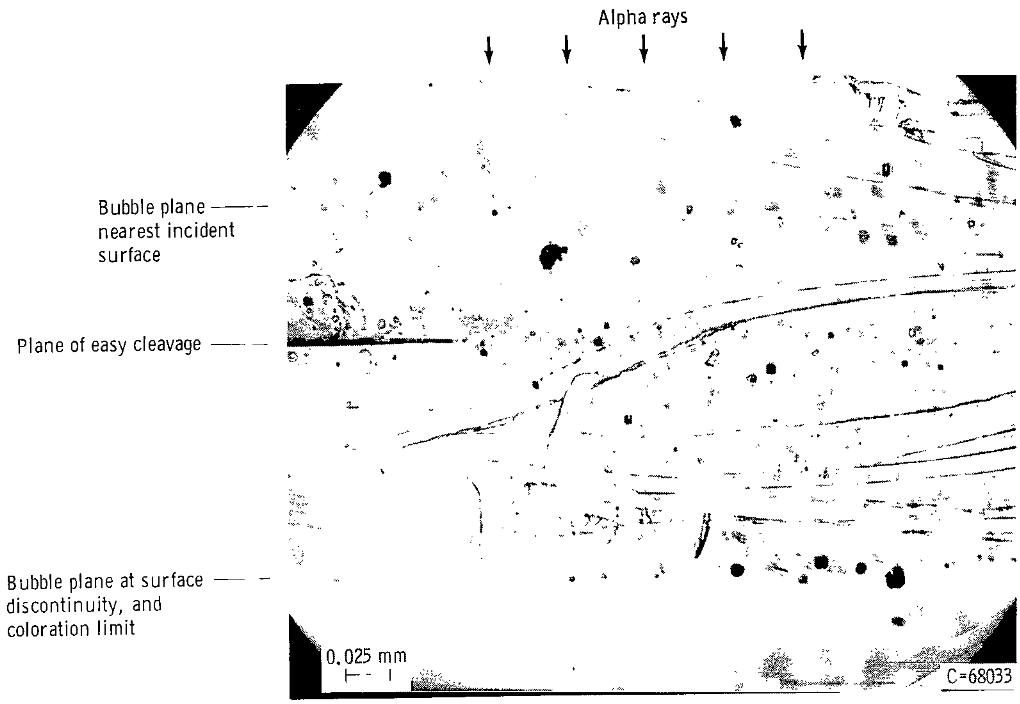


Figure 2. - Light-scattering plane in a typical specimen.



(a) Left half; not heated.

Figure 3. - Two halves of crystal cleaved normal to damage plane after irradiation.



(b) Right half; heated to 400° C.

Figure 3. - Concluded. Two halves of crystal cleaved normal to damage plane after irradiation.

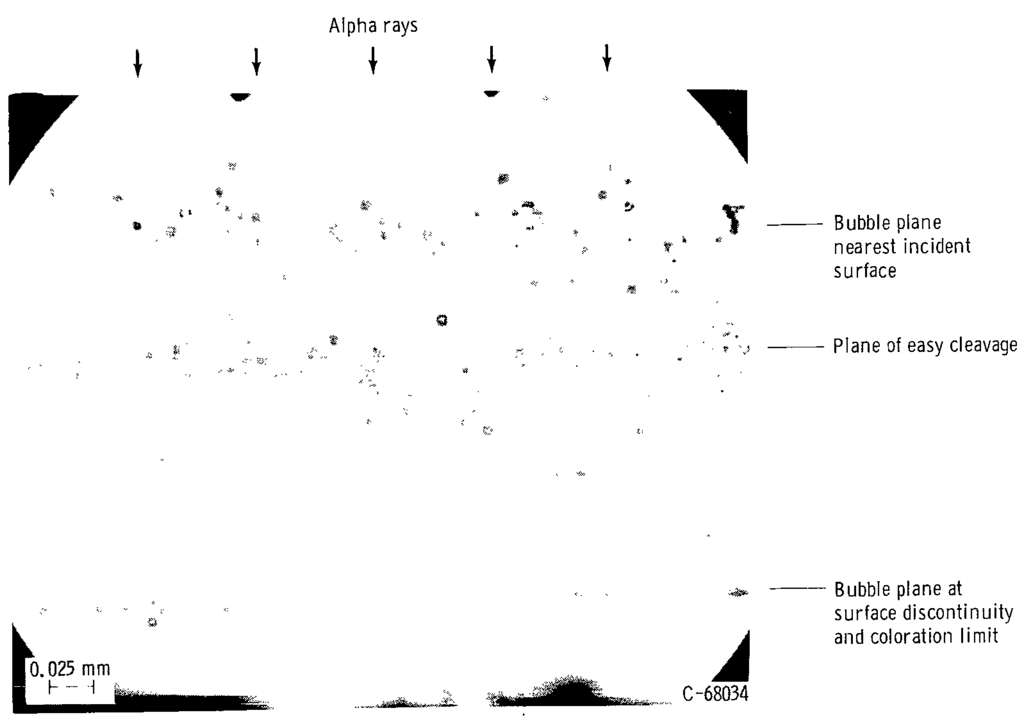


Figure 4. - Planar regions containing cavities. Focal plane about 0.1 millimeter below surface.

continuity, the middle one corresponds to the plane of easy cleavage, and the top one is a poorly defined region nearest the incident surface. Figure 4 shows the same cavity regions at a focal plane about 0.1 millimeter below the surface. Here the cavity planar regions are well defined and more heavily populated. The bottom and the middle regions are narrow and may be characterized loosely as planes. The top region is comparatively broad, quite poorly defined, at a depth of 0.48 millimeter from the surface exposed to the alpha beam, and about 0.06 millimeter thick. The middle cavity region (0.02 mm thick), which appears to be most densely populated, contains the plane of easy cleavage at a depth of 0.56 millimeter. The bottom cavity region with a thickness of 0.01 millimeter is at the plane of surface cleavage discontinuity at a depth of 0.72 millimeter. In addition to these planar regions, there are cavities throughout the band between 0.48 and 0.72 millimeter that tend to be formed predominantly near the middle of the band.

It can be seen from figures 3(b) and 4 that these cavities are rectangular (most of which are cubic) rather than spherical. That the orientation of the individual cavities is along the crystal axes is shown in figure 5. A (111)

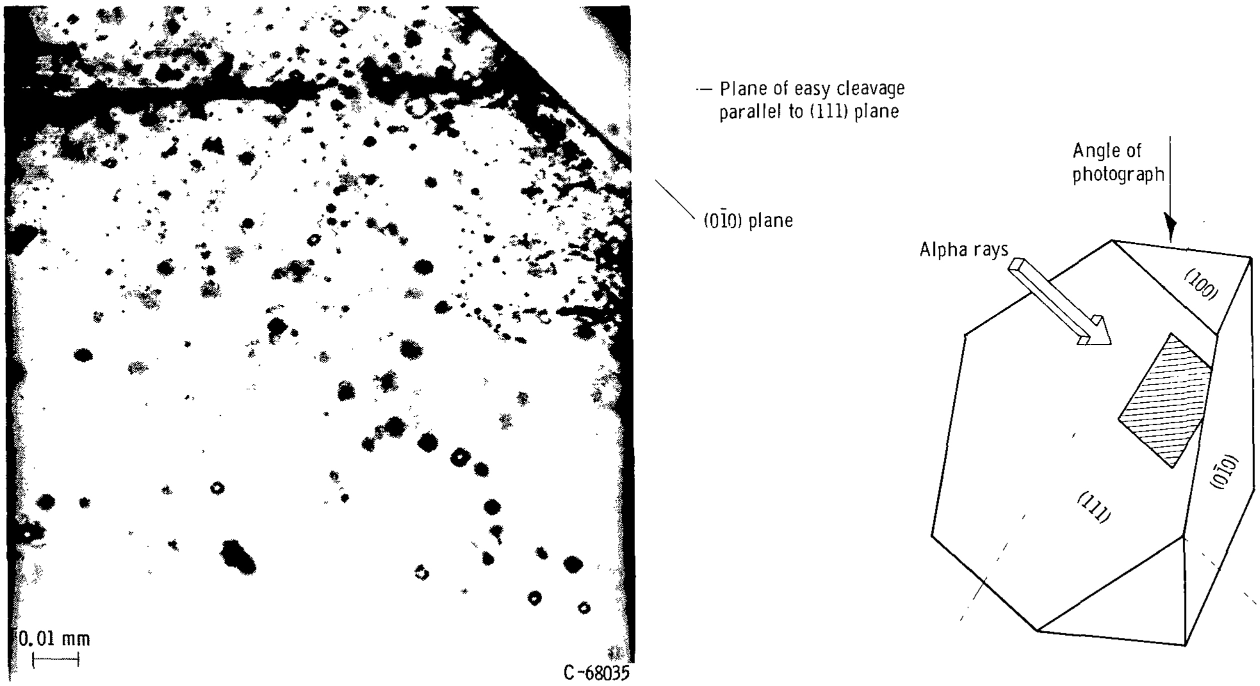


Figure 5. - Photomicrograph of sodium chloride, irradiated on the (111) plane, with focal plane parallel to (100) plane. Total flux, 5×10^{15} particles per square centimeter.

surface was prepared by sawing and polishing the crystal, which was bombarded with the alpha beam normal to the (111) surface and finally bleached. The damaged planar region is parallel to the (111) plane. The view in figure 5 is looking down on the (100) plane with the crystal axes rotated 45° in the plane of the figure. The direction of the incident beam can be envisioned by imagining an arrow placed in the plane of the figure that points to the top of the page, and then imagining that the tail of the arrow is raised from the page at an angle of 45°. The dark horizontal line near the top of the photograph is the intersection of the damaged plane (111) and the focal plane (100). The cavities at the top of the photograph lie above the focal plane; those at the bottom lie below it. The individual cavities are oriented along the cubic crystal axes.

Helium Gas Measurements

The helium leak detector indicated that helium was present in the colored crystals but that it escaped when the samples were crushed. No helium was detected when the bleached samples were crushed.

An irradiated colored crystal was heated in the vacuum chamber, and the leak detector indicated that helium gas escaped as the crystal was heated and bleached. The escape of detectable helium was nearly complete when the temperature reached about 325° C, at which time the bleaching was also complete.

Chlorine and Sodium Measurements

A crystal bombarded with 5×10^{15} alpha particles per square centimeter was found to have 2.1×10^{18} free chlorine atoms per cubic centimeter and 1.6×10^{18} free sodium atoms per cubic centimeter. A crystal bombarded with 1×10^{15} alpha particles per square centimeter had 1.1×10^{18} free chlorine atoms per cubic centimeter and 0.6×10^{18} free sodium atoms per cubic centimeter. The measured values for free chlorine and sodium atoms in a bleached crystal returned to the values for an unirradiated crystal. This indicates that the bleaching was complete and that the crystal returned to a stoichiometric balance.

DISCUSSION

Surface Cleavage Pattern

The microphotographs of freshly cleaved surfaces normal to the damaged plane reveal a sharp discontinuity in the surface cleavage pattern at the limit of the colored region. This discontinuity is probably caused by the abrupt change in dislocation density between the irradiated and the unirradiated portions of the crystal. The dislocation density is known to affect the surface cleavage pattern, and the increase in hardness of alpha-particle-irradiated crystals has also been ascribed to the increased dislocation density (ref. 9).

Easy Cleavage Plane

The occurrence of the easy cleavage plane can be explained qualitatively by the fact that the heaviest damage in the crystal occurs near the end of the beam path. When the alpha-particle flux is greater than about 10^{15} alpha particles per square centimeter, the total damage in the region nearest the end of the particle path is sufficient to harden the crystal and produce clustered defects. Evidence for clustering of defects and for lattice strain after exposure to 10^{18} neutrons per square centimeter in lithium fluoride has been observed in X-ray studies of irradiated crystals (ref. 10). The density of ionization at the end of the alpha-particle path in sodium chloride is the same as the volume of ionization of lithium fluoride exposed to 10^{18} neutrons per square centimeter; the damage in the two cases is comparable. Tapping the crystal with a blunt object stresses it somewhat randomly and causes it to separate in the region of heavy damage.

The position of the easy cleavage plane cannot be predicted from the preceding explanation. The density of ionization along the path is greatest near the end at a depth of about 0.68 millimeter in sodium chloride. This point is beyond the observed positions of the easy cleavage plane, which occur between extreme depths of 0.48 and 0.62 millimeter. Hence, this explanation in terms of the amount of damage is not completely satisfactory.

Crystallographic Cavities

In discussing the phenomenon of the formation of the crystallographic cavities, it is useful to compare the results of the determination of the presence or absence of helium, chlorine, and sodium obtained in this investigation with the results for comparable quantities in lithium fluoride obtained by other investigators. These results are summarized in the following table:

Colored crystals		Source	Bleached crystals		Source
Component	Present or absent		Component	Present or absent	
Helium	Present	This investigation	Helium	Absent	Ref. 5 and this investigation
			Helium aggregates	Present, perhaps	Ref. 10
Fluorine	Present	Ref. 11	Fluorine	Absent	Ref. 11
Chlorine	Present	This investigation	Chlorine	Absent	This investigation
Fluorine aggregates	Present, perhaps	Ref. 10	Fluorine aggregates	Absent	Ref. 10
Lithium aggregates	Present, perhaps	Ref. 10	Lithium aggregates	Absent	Ref. 10
Sodium	Present	This investigation	Sodium	Absent	This investigation
Lithium platelets	Present	Refs. 12 and 13	Lithium platelets (heated to 350° C)	Absent	Refs. 12 and 13
			Colloidal lithium	Present, perhaps	Ref. 5

It is evident that helium and halogen gases are present in the crystal after irradiation. After the annealing process, which gives rise to bleaching and cavity formation, these gases are not present. Reference 10 is in partial with this last statement, but there was available no direct evidence for the presence of helium gas in the crystal after annealing. A similar situation is found, in general, for free alkali metals; free metal atoms are present in the irradiated colored crystals but are absent in the irradiated bleached crystals. An exception to this evidence is given in reference 5, where it is supposed that colloidal lithium is present in the crystal after annealing. This evidence is based on optical transmission measurements and is reasonable, but somewhat indirect, evidence.

The general agreement of these results for sodium chloride and lithium fluoride makes it reasonable to consider the suggestions for the source of the cavities or voids in lithium fluoride as they might apply to alpha-irradiated sodium chloride. In reference 14, where much of the experimental work on lithium fluoride is reviewed, the authors discuss the possibility that the coagulation of vacancies is responsible for the crystallographic cavities. One process suggested for vacancy formation is coagulation of the anion vacancies left by fluorine that escapes from the crystal. If this were the case, metallic lithium would be present in the crystal. The results of the present experiment on sodium chloride rule out the possibility that anion vacancies in sodium chloride, left by chlorine that escaped, coagulate to form the cavities, because no sodium metal is found in the bleached crystals. An alternative suggestion is that neutral cation-anion vacancy pairs coagulate to form the voids. The present results show that the coagulation of neutral cation-anion vacancies could be responsible for forming the cavities. It may not be necessary for anion-cation vacancy pairs as such to coagulate provided that both anion and cation vacancies contribute to the cavities. The problem here is in deciding what happens to the excess sodium metal, since it can be assumed that the chlorine migrates to the surface and escapes. Possibly, the sodium also migrates to the surface and forms a layer of oxide or carbonate by reaction with the ambient gas, or forms a layer of chloride with free chlorine. Another suggestion is that, in lithium fluoride, the voids are formed by helium gas capturing vacancies and expanding into gas bubbles. In the present sodium chloride crystals, the annealed specimens containing cavities did not contain helium gas, which, therefore, seemed to rule out this process. It is also possible, however, that the helium gas plays a role in the vacancy coagulation but escapes during the annealing process; the measurements cannot rule out this possibility.

In alpha-irradiated sodium chloride, the cavities form in planar regions parallel to the incident surface, while in neutron-irradiated lithium fluoride, they are formed first along subboundaries throughout the specimen. The lithium fluoride crystals were annealed at temperatures above 600° C, in some cases for periods of an hour or more. The cavities appeared in sodium chloride crystals after the temperature was raised to 400° C for as little as 5 minutes. The location of the cavities in planes in sodium chloride may be partly due to the short annealing times, and these particular formations may be an initial stage in the process. Planes of cavities seem to form at the coloration limit, at the plane of easy cleavage, and sometimes at other apparently random positions where they are usually not so sharply defined. It may be that the cavities in sodium chloride grow by the capture of vacancies and that the coloration limit and plane of easy cleavage are major sources of vacancies. The density of cavities is greatest at the easy cleavage plane with numerous voids on both sides of the plane (see e.g., fig. 4, p. 6).

A study of copper that was irradiated with 30-Mev alpha particles gives some information on the distribution of bubbled regions in incompletely annealed specimens. In these experiments, bubbles of crystallographic shape were observed in a narrow band at the end of the alpha-particle range. The explanation given in reference 3 is that the helium gas injected by the bombardment attempts to precipitate in the form of gas bubbles when the metal is heated and captures large numbers of vacancies to acquire the necessary extra space. The gas bubbles first appear at given boundaries and at the periphery of the band. These were taken to be vacancy sources for bubble formation. Annealing for longer periods of time caused the bubbled zones to spread farther out from the vacancy sources and eventually fill the whole band with bubbles. The conclusion was that the bubbles grow by capture of vacancies and not by migration of helium atoms to the vacancy sources, since the rate of advance of the bubbled zones is greatest near the periphery of the band, where the concentration of helium is lowest, and least in the center of the band, where most of the helium is deposited.

Hence, for both lithium fluoride and copper, the distribution of the crystallographic cavities or bubbles is dependent on the annealing time. It seems probable that this will be true for sodium chloride also. In copper, the location of the cavities is at the end of the range of the alpha particles; this is not the case in sodium chloride. The range of the alpha particles will be normally distributed about the mean range R, with a standard deviation given by $\alpha_0/\sqrt{2}$, where α_0 is the range-straggling parameter for a monoenergetic beam. Essentially, all the ranges will be included within a spread of $4\alpha_0$. This range spread can be increased to $4\alpha_0\sqrt{2}$ in order to take some account of the effects of a spread in energy of the beam. A good approximation (ref. 15) for α_0 is that $\alpha_0 \simeq 0.015\ R$, while an upper limit for R given by the coloration limit is 0.72 millimeter. The maximum spread in range is given by 0.06 millimeter, which is much smaller than the observed variation in position of the crystallographic cavities, and much smaller than the variation in position of the easy cleavage plane. Thus, the hypothesis that helium gas plays a role in the formation of the cavities requires the gas to diffuse in sodium chloride much more readily than in copper. This is actually a reasonable supposition since sodium chloride has a more open structure than copper. If the helium gas does cause or aid the cavity growth, the shorter time required for the cavity formation in sodium chloride than in copper is in agreement with a higher diffusion rate in sodium chloride.

SUMMARY OF RESULTS

The experimental results of bombarding sodium chloride crystals with 40-Mev alpha particles are as follows: The irradiated crystals are colored throughout the rectangular region between the incident face of the crystal and a plane parallel to this surface at a depth of 0.73±0.01 millimeter. The depth of the colored region, which is 0.72 millimeter, agrees well with the computed range of the alpha particles. In addition to the colored region, there is a damaged planar region normal to the alpha beam nearest the end of the particle range. For fluxes greater than 10^{14} alpha particles per square centimeter, the damaged planar region is characterized by (1) a line of discontinuity of the surface cleavage pattern (on surfaces normal to the damaged plane) at the coloration limit, (2) a plane of easy cleavage that occurs at depth between 0.43 and 0.62 millimeter, and (3) crystallographic cavities that appear after heating the crystal to 400° C for about 5 minutes. The plane of easy cleavage occurs in the region of heavy damage where the density of ionization is comparable to that produced by 10^{18} neutrons per square centimeter, at which flux lithium fluoride crystals become friable. These crystallographic cavities usually are concentrated at the plane of easy cleavage, at the coloration limit, and sometimes in other planar regions normal to the incident-beam direction. The 5-minute heating suffices to bleach completely the color centers produced by the irradiation. During this time the helium gas from the stopped alpha particles escapes from the crystals. Helium is present in the irradiated colored crystals but it is not present in the irradiated bleached crystals. Some free chlorine and sodium atoms are present in the irradiated colored crystals, but the irradiated bleached crystals contain no free chlorine or sodium.

CONCLUSIONS

In view of these experimental results, the following conclusions about the formation of the damaged planar region in sodium chloride crystals are drawn: The coagulation or agglomeration of anion and cation vacancies is possibly the cause of the crystallographic cavities. The coagulation of anion vacancies alone does not cause the cavities. The agglomeration of injected alpha particles into helium gas is consistent with the present result if the gas diffuses more readily in sodium chloride than in copper or lithium fluoride.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, February 17, 1964

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68

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